DOI: https://doi.org/10.24425/amm.2023.141470

YEON-JOO LEE©^{1,2}, DO-HUN KWON©¹, EUN-JI CHA©¹, YONG-WOOK SONG©², HYUN-JOO CHOI©², HWI-JUN KIM©¹*

EFFECT OF COOLING RATE ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Al-33 WT.% Cu ALLOY

Directed energy deposition (DED) is an additive manufacturing process wherein an energy source is focused on a substrate on which a feedstock material is simultaneously delivered, thereby forming a small melt pool. Melting, solidification, and subsequent cooling occur at high rates with considerable thermal gradients compared with traditional metallurgical processes. Hence, it is important to examine the effects of cooling rates on the microstructures and properties of the additive manufactured materials. In this study, after performing DED with various energy densities, we investigated the changes in the microstructures and Vickers hardness of cast Al-33 wt.% Cu alloy, which is widely used to estimate the cooling rate during processing by measuring the lamellar spacing of the microstructure after solidification. The effects of the energy density on the cooling rate and resultant mechanical properties are discussed, which suggests a simple way to estimate the cooling rate indirectly. This study corresponds to the basic stage of the current study, and will continue to apply DED in the future.

Keywords: Laser Melting; Cooling rate; Lamellar Spacing; Hardness; Al-33 wt.% Cu alloy

1. Introduction

Aluminum alloys have attracted increasing attention as one of the lightweight metals because of their abundant malleability, excellent corrosion resistance, and high thermal as well as electrical conductivities [1]. Because of these excellent characteristics, they are widely used in construction equipment's, automobile, aerospace, sport/leisure goods, electric, electronic as well as information technology industries [2].

Casting is one of the most frequently used manufacturing processes for aluminum alloys in various industries because of its high-fabrication speed and low restrictions on the size and weight of products [3]. Nevertheless, because of uneven grain and precipitation sizes, pores and segregation are likely to occur, which can lead to deterioration in mechanical properties. Additionally, it has the disadvantage of low-dimensional precision, limitation in manufacturing thin products, and causing pollution. Additive manufacturing (AM) can be used to manufacture complex-shaped parts because of its high degrees of freedom in design compared to the existing traditional processes [4]. Nevertheless, the application to AM processes has been limited because of their high-thermal conductivities and refractive indexes with occasionally accompanying large freezing ranges. Particularly, during directed energy deposition (DED) – type AM, the cooling rates lie in a wide range from 10^3 to 10^8 K/s [5]. Thus, efforts have been made to measure the cooling rates and control thermal histories during AM to manufacture aluminum alloys with minimized thermal cracks or unwanted defects [6]. Although previous studies have investigated the effects of thermal history, such as the temperature difference and cooling rate during AM on the microstructure, residual stress, volume defects, and mechanical properties of the material by repeated melting and fast solidification, which in turn affects mechanical properties [7], in-situ measurement of cooling rates during AM has not been well studied.

Al-33 wt.% Cu alloy has a eutectic composition and exhibits a lamellar structure upon solidification and is cooled. Using these characteristics, the cooling rate during the process can be calculated by measuring the lamellar spacing through observation of the microstructure of Al-33 wt.% Cu [8-10]. In this study, lasers with various energy densities were irradiated to the casted Al-33 wt.% Cu plate by changing the laser power and scanning

Corresponding author: khj@kitech.re.kr



^{© 2023.} The Author(s). This is an open-access article distributed under the terms of the Creative Commons Attribution-NonCommercial License (CC BY-NC 4.0, https://creativecommons.org/licenses/by-nc/4.0/deed.en which permits the use, redistribution of the material in any medium or format, transforming and building upon the material, provided that the article is properly cited, the use is noncommercial, and no modifications or adaptations are made.

¹ KOREA INSTITUTE OF INDUSTRIAL TECHNOLOGY, RESEARCH INSTITUTE OF ADVANCED MANUFACTURING & MATERIALS TECHNOLOGY, 156, GAETBEOL-RO, YEONSU-GU, INCHEON, REPUBLIC OF KOREA 21999

² KOOKMIN UNIVERSITY DEPT. OF ADVANCED MATERIALS ENGINEERING, SEOUL, KS013, REPUBLIC OF KOREA

speed. The cooling rate was calculated on the surface of the Al-33 wt.% Cu plate irradiated with various energy densities and changes in the behavior of the microstructure and mechanical properties were observed.

2. Experimental

We investigated the changes in hardness and microstructure of cast Al-33 wt.% Cu alloy after irradiation at various energy densities. Master alloy was first prepared by vacuum plasma melting to prepare a laser-irradiated substrate, and then, the plate was manufactured by vacuum centrifugal casting. The microstructure of the cast plate was observed after cutting 2 mm off from the surface to eliminate microsized surface pores. The plates were sand blasted to produce rough surfaces to suppress the reflection of the laser and the oxide film on the surface was removed by immersion in 1 M NaOH aqueous solution for 10 s before irradiation. To irradiate lasers with various energy densities, a total of nine laser irradiation conditions were used by changing the laser power (P) to 500, 700, and 900 W and the laser scanning speed (S) to 300, 1000, and 1500 mm/min.

To calculate the laser density and cooling rate after laser treatment at various energy densities, the microstructure of the molten part was observed using an optical microscope (Nikon-ECLIPSE-MA200, Japan) and a scanning electron microscope (FEI-SirionTM-XFLASH6160, the Netherlands). To observe the microstructure and molten area, laser-treated Al-33 wt.% Cu plate cross-sections were mechanically polished using sandpaper (#80-#2000) and 0.5 μ m diamond abrasive after mounted using polycoat. The lamellar structure of samples was clearly observed by etching in a solution of HNO₃-HCl-HF-DI water (= 5:3:2:190) for 15 s. The lamellar spacing was measured more than 10 times and averaged, and the cooling rates were calculated. The Vickers hardness tests were conducted to observe the mechanical properties under 2.942 N indenter load for 10 s.

3. Results and discussion

Fig. 1 (a) and (b) show the macrostructures and lamellar structure, respectively, in the cross-sectional images of the Al-33 wt.% Cu plate after the laser melting process. The average lamellar spacing of the Al-33 wt.% Cu as-cast plate was 382.62 nm, it was confirmed that the lamellar spacing after irradiation (123.77 nm) decreased by 0.3 times or more on. A schematic showing the depth (d), width (w) and area (A) of the molten area of the Al-33 wt.% Cu plate, as well as the radius (r)

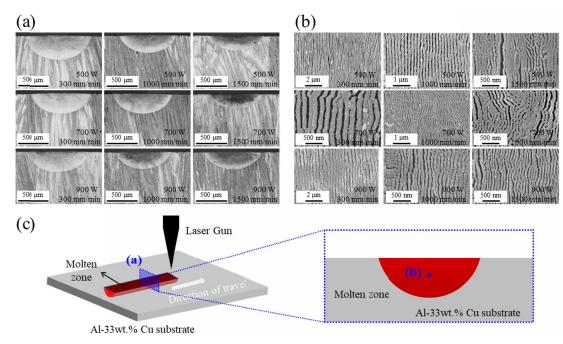


Fig. 1. Cross-sectional images of Al-33 wt.% Cu plate after laser melting process. (a) Macrostructures, (b) lamellar structure with irradiating conditions and (c) schematic diagram of observation positions of (a) and (b)

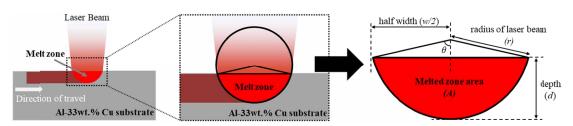


Fig. 2. Schematic of the laser melt pool for calculating laser energy density

Р	S	W	d	r	Θ	Α	λ	R
[W]	[mm/min]	[µm]	[µm]	[µm]	[°]	[µm ²]	[nm]	[K/s]
500	300	1126.2	399.4	596.7	70.7	327948.5	188.1	6.84×10^{3}
500	1000	976.3	300.7	546.6	63.3	209683.5	112.0	1.93×10 ⁴
500	1500	915.1	248.5	545.5	57.0	160050.9	84.2	3.41×10 ⁴
700	300	1045.3	355	562.2	68.4	268711	175.6	7.85×10^{3}
700	1000	1030.5	357	550.3	69.4	267219.5	86.3	3.25×10^4
700	1500	927	265.2	537.6	59.6	174018.2	105.3	2.18×10 ⁴
900	300	1094.6	388.5	579.8	70.7	310087.7	205.4	5.74×10 ³
900	1000	978.3	298.8	549.8	62.8	208560.3	91.1	2.92×10 ⁴
900	1500	979.2	307.6	543.4	64.3	215691.7	66.0	5.56×10 ⁴

Experimental results with laser melting parameters

and angle (θ) of the laser beam is shown in Fig. 2. The laser was assumed to be a complete sphere, and the laser radius and the size of the molten area could be calculated using the measured depth and width of the molten area. TABLE 1 lists the width (w) and depth (d) of the molten area, as well as the calculated radius of laser beam (r), angle (θ), molten area (A), average lamellar spacing (λ), and cooling rate (R).

Eq. (1) was used for calculating the cooling rate through the lamellar spacing by referring to the research of R.M. Srivastava [11].

$$R = \frac{\Delta H_f}{c_p} \frac{3K}{\lambda^2} \tag{1}$$

Here, *R* is the cooling rate [K/s], λ is lamellar spacing [nm], c_p and ΔH_f are the specific heat thermal coefficients, and $\Delta H_f/c_p = 440$ K in the Al-Cu system. The constant $K = 27.5 \times 10^{-12}$ cm³s⁻¹ is determined for the Al–Al₂Cu eutectic system. The cooling rate of Al-33 wt.% Cu as-plate was calculated as 1.67×10^3 K/s. It was confirmed that the cooling rate of all the specimens increased from 3.4 times to 33.3 times after the laser treatment. The laser density was calculated to observe the direct effect of the laser on the Al-33 wt.% Cu plate. Laser density was calculated by Eq. (2). [12].

$$E = \frac{P}{S \cdot r} \tag{2}$$

Here, *E* is the laser density [J/mm²], *P* is the laser power [W], *S* is the laser scanning speed [mm/min], and *r* is the radius of the laser beam [μ m]. In this study, the laser density was calculated using the laser radius *r* value shown in Table 1. The difference from the experimental value was less than 10% when the theoretical laser radius value (1000 μ m) was substituted into the *r* value. The results calculated in this way are shown in Fig. 3 by color according to the range of laser density. It was confirmed that the higher the laser power and the slower the laser scanning speed, the higher the laser density. It was also confirmed that various laser densities were applied to the Al-33 wt.% Cu plate from a minimum of 34.7 to a maximum of 307.2 J/mm². Fig. 4 shows the Vickers hardness of the Al-33 wt.% Cu plate before and after laser irradiation according to the laser energy density.

It was confirmed that the Vickers hardness increased in all samples after laser irradiation. The hardness was increased from at least 1.16 times to a maximum of 1.57 times than before laser treatment (182 HV), and the maximum hardness was 285 HV when the energy density was 65.90 J/mm² (power 500 W, scan speed 1500 mm/min.). The cooling rate is affected by the laser density that changes depending on the laser power and the laser scan speed [13]. When a laser is irradiated to the surface of the casting material, the Al-33 wt.% Cu plate acts as a heat sink, and only the region where the laser is directly irradiated melts, such that a cooling rate faster than that of the casting process is obtained. Additionally, since the entire specimen is not melted, small pores present on the surface of the casting material are removed due to the remelting effect while maintaining the shape of the specimen [14]. Thus, hardness could be improved in all samples subjected to laser treatment. If the laser density is low, pores and irregular molten areas are formed because of the nonmelting metal. However, if the laser density applied to the material is high, the cooling rate after melting is too high to discharge steam generated during the melting process and remains in the melting ground to form pores [15]. Thus, an appropriate laser density should be applied to maximize the hardness through

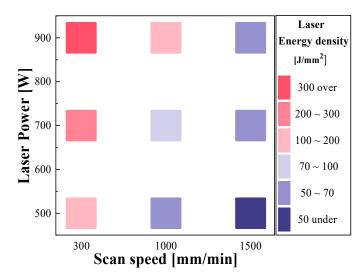


Fig. 3. Laser energy density map with varying laser power and scan speed

the removal of pores and miniaturization of the microstructure depending on the material, and it was verified that the maximum hardness is achieved at a laser density of 65.90 J/mm². Laser density for each power and scanning speed can be experimentally verified through laser surface remelting using an Al-33 wt.% Cu material capable of measuring the cooling rate. In future work, the proposed method is expected to reduce pores and effectively increase hardness in very wide or local areas that cannot be accessed by other treatment methods.

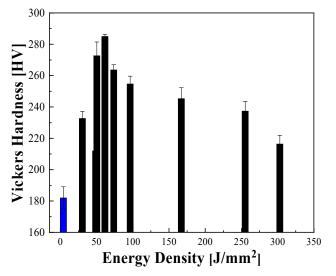


Fig. 4. The variation of Vickers hardness values of irradiated Al-33 wt.% Cu plate with varying laser energy density

4. Conclusions

In this study, by irradiating lasers with various energy densities onto the Al-33 wt.% Cu cast plate, changes in the microstructure and Vickers hardness were investigated for the samples DED treated with energy densities, varied from 34.7 to 307.2 J/mm². The hardness (212–285 HV) of the Al-33 wt.% Cu plate was significantly improved after irradiating a laser using

DED by at least 14% to a maximum of 36% compared to that (182 HV) of the as-cast plate. Using the laser melting process, mechanical properties are expected to improve by controlling the microstructure through grain refinement by providing a high cooling rate.

REFERENCES

- L. Wzorek, R. Wolniak, K. Lyp-wronska, M. Wiewiora, P. Noga, A. Wzorek, Arch. Metall. Mater. 63, 667-672 (2018).
- [2] S.T. Mavhungu, E.T. Akinlabi, M.A. Onitiri, F.M. Varachia, Procedia Manuf. 7, 178-182 (2017).
- [3] R. Wladysiak, A. Kozun, T. Pacyniak, Arch. Metall. Mater. 62, 187-194 (2017).
- [4] N. T. Aboulkhair, M. Simonelli, L. Parry, I. Ashcroft, C. Tuck, R. Hague, Prog. Mater. Sci. 106, 100578 (2019).
- [5] M.J. Zadeh, G.P. Kumar, P.S. Branicio, M. Seifi, J.J. Lewandowski, F. Cui, J. Funct. Biometals 9, 1-32 (2018).
- [6] M. Kohler, L. Sun, J. Hensel, S. Pallaspuro, J. Komi, K. Dilger, Z. Zhang, Mater. Design 210, 110122 (2021).
- [7] J. Piatkowski, A. Grabowski, M. Czerepak, Metall. Mater. 16, 217-221 (2016).
- [8] C.S. Tiwary, D. R. Mahapatra, K. Chattopadhyay, Appl. Phys. Lett. 101, 171901 (2012).
- [9] T. Koziel, Arch. Metall. Mater. 60, 767-771 (2015).
- [10] M. Zimmermann, M. Carrard, W. Kurz, Acta Metall. 37, 3305-3313 (1989).
- [11] R.M. Srivastava, J. Eckert, W. Loser, B.K. Dhindaw, L. Schultz, Mater. Trans. 43, 1670-1675 (2002).
- [12] S. Webster, K. Ehmann, J. Cao. Procedia Manuf. 48, 691-696 (2020).
- [13] M. Gorny, G. Sikora, Arch. Foundry Eng. 14, 21-24 (2014).
- [14] C. Qui, N.J.E. Adkins, M.M. Attallah, Mater. Sci. Eng. A 578, 230-239 (2013).
- [15] I. Jung, J. Choe, J. Yun, S. Yang, D. Yang, Y. Kim, J. Yu, Arch. Metall. Mater. 64, 571-578 (2019).